
सल्फ़ानिलिक एसिड, तकनीकी — विशिष्टि
(तीसरा पुनरीक्षण)

**Sulphanilic Acid, Technical —
Specification**
(*Third Revision*)

ICS 71.080.40

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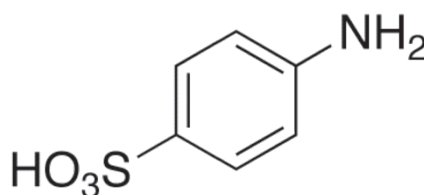


भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Sulphanilic acid is an important organic intermediate used in organic synthesis and in the production of quinone. It is also widely used in the manufacture of variety of dyes and drugs. It is also known as *p*-aminobenzenesulphonic acid and 4-aminobenzenesulphonic acid and is represented by the following structural formula:



SULPHANILIC ACID

(MOLECULAR MASS = 173.2)

(CAS REGISTRY NO.: 121-57-3)

This standard was first issued in 1964 and subsequently revised in 1973 and 1986. The first revision was taken up to revise the limit of assay of sulphanilic acid and matter insoluble in sodium carbonate. With further progress in the dyestuff industry and on the basis of the latest development in the industry the Committee decided to undertake the second revision of this standard. The second revision included changes in the limits of assay of sulphanilic acid and aniline content. Characteristic of impurities such as orthanilic acid, metanilic acid, and aniline-2,5-disulphonic acid were also incorporated.

In this (third) revision the requirements of assay and aniline are once again revised keeping in mind the advancement in industries. The limits of impurities, such as orthanilic acid, metanilic acid, and aniline-2,5-disulphonic acid has been stipulated. Further, test method for determination of impurities like orthanilic acid, metanilic acid, and aniline-2,5-disulphonic acid has been updated to HPLC. HPLC method for determination of assay of sulphanilic acid has also been incorporated.

The composition of the Committee responsible for formulation of this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SULPHANILIC ACID, TECHNICAL — SPECIFICATION

(*Third Revision*)

1 SCOPE

This standard prescribes the requirement, methods of sampling and testing for sulphanilic acid, technical.

2 REFERENCES

The following standards contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (<i>third revision</i>)
5299 : 2001	Methods for sampling and tests for dye intermediates (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of grey paste or powder free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable containers like jute bags or drums (*conforming to IS 2552*) lined with polyethylene film.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of manufacturer and his recognized trade-mark, if any;
- c) Net mass of material;
- d) Month and year of the manufacture;
- e) Batch or lot number; and
- f) Any other statutory requirements.

4.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of preparing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Tests for determination of assay shall be conducted on each of the individual samples.

5.2.2 Test for the determination of the remaining characteristics, namely, aniline content, matter insoluble in sodium carbonate solution, ash, orthonilic acid, metanilic acid and aniline-2,5-disulphonic acid content shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 For Individual Samples

The lot shall be declared as conforming to the requirement of assay, if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 For Composite Samples

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (**5.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

Table 1 Requirements for Sulphanilic Acid, Technical
(Clauses 3.2, 5.3.1, 5.3.2 and 6.2)

SI No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex	Clause No. in IS 5299
(1)	(2)	(3)	(4)	(5)
i)	Assay by nitrite value, percent by mass, <i>Min</i>	98.00	A	—
	or Assay by HPLC ¹⁾ , percent by mass, <i>Min</i>		B	—
ii)	Matter insoluble in sodium carbonate solution, percent by mass, <i>Max</i>	1.0	—	11.2
iii)	Ash content, percent by mass, <i>Max</i>	1.0	—	12.1
iv)	Aniline content, percent by mass, <i>Max</i>	0.2	C	—
v)	Metanilic acid and aniline-2,5-disulphonic acid, percent by mass, <i>Max</i>	0.5	C	—
vi)	Orthanilic acid, percent by mass, <i>Max</i>	1.0	C	—

¹⁾ In case of disputes, assay by HPLC shall be the referee method.

6 TEST METHODS

6.1 Preparation of Sample

Mix the sample well by rotating the bottle several times. Transfer a portion immediately into a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acid or alkaline fumes. Dry the sample at 105°C to constant mass and use this prepared sample for tests.

6.2 Tests shall be carried out on the prepared sample (**6.1**), according to the methods prescribed in col 4 and col 5 of Table 1.

6.3 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, SI No. (i)]

DETERMINATION OF SULPHANILIC ACID CONTENT (ASSAY) BY NITRITE VALUE

A-1 REAGENT

A-1.1 Concentred Hydrochloric Acid

A-1.2 Potassium Bromide

A-1.3 Standard Sodium Nitrite Solution, 0.1 N.

A-1.4 Potassium Starch Iodide Paper

A-2 PROCEDURE

Weight accurately 0.7 g sample in 1 l beaker containing 50 ml sodium carbonate solution. Add about 200 ml of water, 35 ml of hydrochloric acid, 5.0 g of potassium bromide and washed-ice. Cool to 10 to 15°C. Titrate with standard sodium nitrite solution with constant

stirring mechanically, until a distinct blue ring on the starch potassium iodide paper indicates the end point. The end point should persist for 10 min without further addition of the nitrite solution.

A-3 CALCULATION

Assay (by nitrite value), percent by mass =

$$\frac{V_1 \times N_1 \times 319}{M}$$

where

V_1 = volume of standard sodium nitrite solution used in the titration, ml;

N_1 = normality of sodium nitrite solution; and

M = mass of the material taken, g.

ANNEX B

[Table 1, SI No. (i)]

DETERMINATION OF SULPHANILIC ACID CONTENT (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

B-1 OUTLINE OF THE METHOD

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry to identify, quantify or purify the individual components of the mixture.

B-2 APPARATUS

B-2.1 Binary gradient liquid chromatography system, with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-2.2 Column

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent

B-3 REAGENT

B-3.1 Acetonitrile, HPLC grade.

B-3.2 Water, HPLC grade.

B-3.3 Sodium Dihydrogen Orthophosphate, HPLC grade.

B-3.4 Tetra-*n*-butylammonium Bromide, HPLC grade.

B-3.5 Sulphanilic Acid, known purity.

B-4 STANDARD PREPARATION

Weigh accurately 0.050 g sulphanilic acid in 100 ml volumetric flask. Dissolve it in water : acetonitrile (20:80) and make it up to the mark with water : acetonitrile (20:80).

B-5 SAMPLE PREPARATION

Weigh accurately 0.050 g sample in 100 ml volumetric flask. Dissolve it in water : acetonitrile (20:80) and make it up to the mark with water : acetonitrile (20:80).

B-6 BUFFER PREPARATION

Take 5.000 g tetra-*n*-butylammonium bromide and 1.666 g sodium dihydrogen orthophosphate in 1 l volumetric flask. Add 200 ml HPLC grade water and completely dissolve it. Make it up to the mark with HPLC grade water.

B-7 FLOW RATE, 1.00 ml/min.

B-8 MOBILE PHASE

Time	Acetonitrile	Buffer
0.01	15	85
12.00	65	35
14.00	65	35
14.01	15	85
20.00	Stop	—

B-9 COLUMN OVEN TEMPERATURE, 40°C**B-10 INJECTION VOLUME, 5 µl****B-11 RUN TIME, 20 min****B-12 WAVELENGTH, 240 nm****B-13 PEAK TIME**

Sulphanilic acid : 5.77 min

B-14 CALCULATION

Calculate the peak area of individual constituent pertaining to sulphanilic acid on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure sulphanilic acid.

$$\text{Sulphanilic acid, percent} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where

A_1 = area of standard sulphanilic acid;

V_1 = injection volume of standard sulphanilic acid;

W_1 = weight of standard sulphanilic acid;

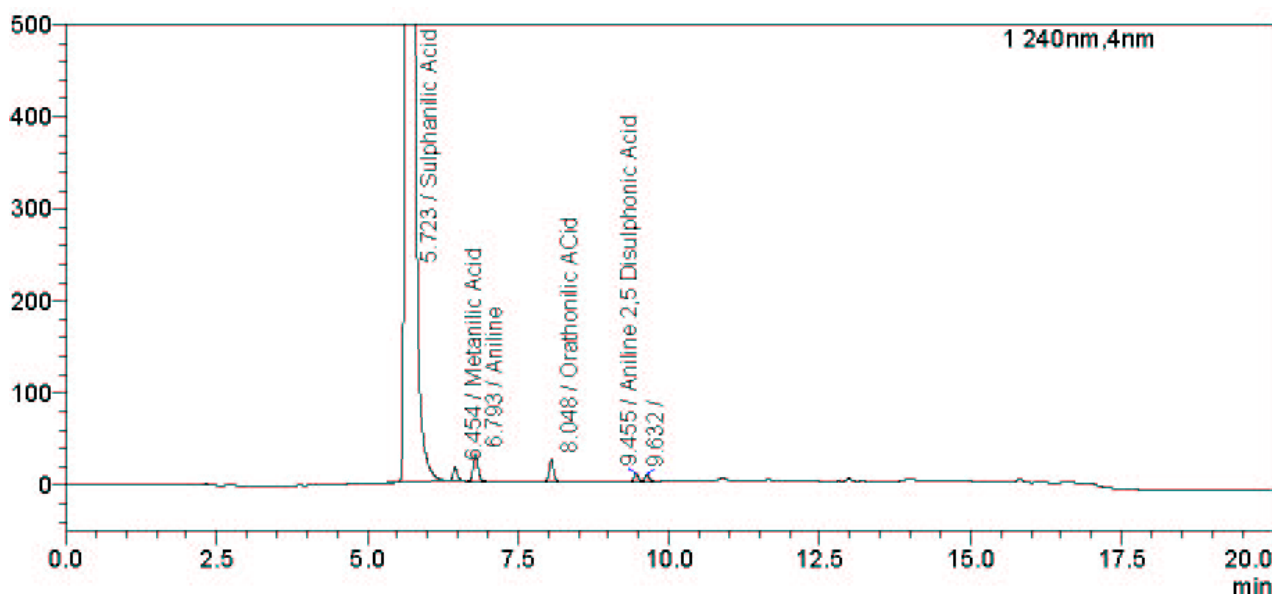
B_1 = total volume of standard sulphanilic acid;

A_2 = area of sulphanilic acid peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.



ANNEX C

[Table 1, Sl No. (iv), (v) and (vi)]

DETERMINATION OF ORTHANILIC ACID, METANILIC ACID, ANILINE CONTENT AND ANILINE-2, 5-DISULPHONICACID

C-1 OUTLINE OF THE METHOD

High-performance liquid chromatography or high pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

C-2 APPARATUS

C-2.1 Binary gradient liquid chromatography system, with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

C-2.2 Column

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent.

C-3 REAGENT

C-3.1 Acetonitrile, HPLC grade.

C-3.2 Water, HPLC Grade.

C-3.3 Sodium Dihydrogen Orthophosphate, HPLC Grade.

C-3.4 Tetra-*n*-butylammonium Bromide, HPLC Grade.

C-3.5 Sulphanilic Acid, known purity.

C-3.6 Metanilic Acid, known purity.

C-3.7 Orthanilic Acid, known purity.

C-3.8 Aniline-2,5-disulphonic Acid, known purity.

C-4 STANDARD PREPARATION

Solution A — Weigh accurately 0.005 g standard aniline in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

Take 1 ml of solution A in 10 ml volumetric flask and dilute it up to the mark with acetonitrile.

Solution B/C/D — Weigh accurately 0.005 g standard metanilic acid/orthanilic acid/aniline-2,5-disulphonic acid in 100 ml volumetric flask dissolve it in water : acetonitrile (20:80) and make it up to the mark with water : acetonitrile (20:80).

Take 1 ml of solution B/C/D in 10 ml volumetric flask. Dilute it up to the mark with water : acetonitrile (20:80).

C-5 SAMPLE PREPARATION

Weigh accurately 0.1000 g sample in 100 ml volumetric flask. Dissolve it in water : acetonitrile (20:80) and make it up to the mark with water : acetonitrile (20:80).

C-6 BUFFER PREPARATION

Take 5.0000 g tetra-*n*-butylammonium bromide and 1.666 6 g sodium dihydrogen orthophosphate in 1 l volumetric flask. Add 200 ml HPLC grade water and completely dissolve it. Make total volume with HPLC grade water.

C-7 FLOW RATE, 1.00 ml/min

C-8 MOBILE PHASE

Time	Acetonitrile	Buffer
0.01	15	85
12.00	65	35
14.00	65	35
14.01	15	85
20.00	Stop	—

C-9 COLUMN OVEN TEMPERATURE, 40°C

C-10 INJECTION VOLUME, 5 µl

C-11 RUN TIME, 20 min

C-12 WAVELENGTH, 240 nm

C-13 PEAK TIME

Sulphanilic acid : 5.72 min

Metanilic acid : 6.45 min

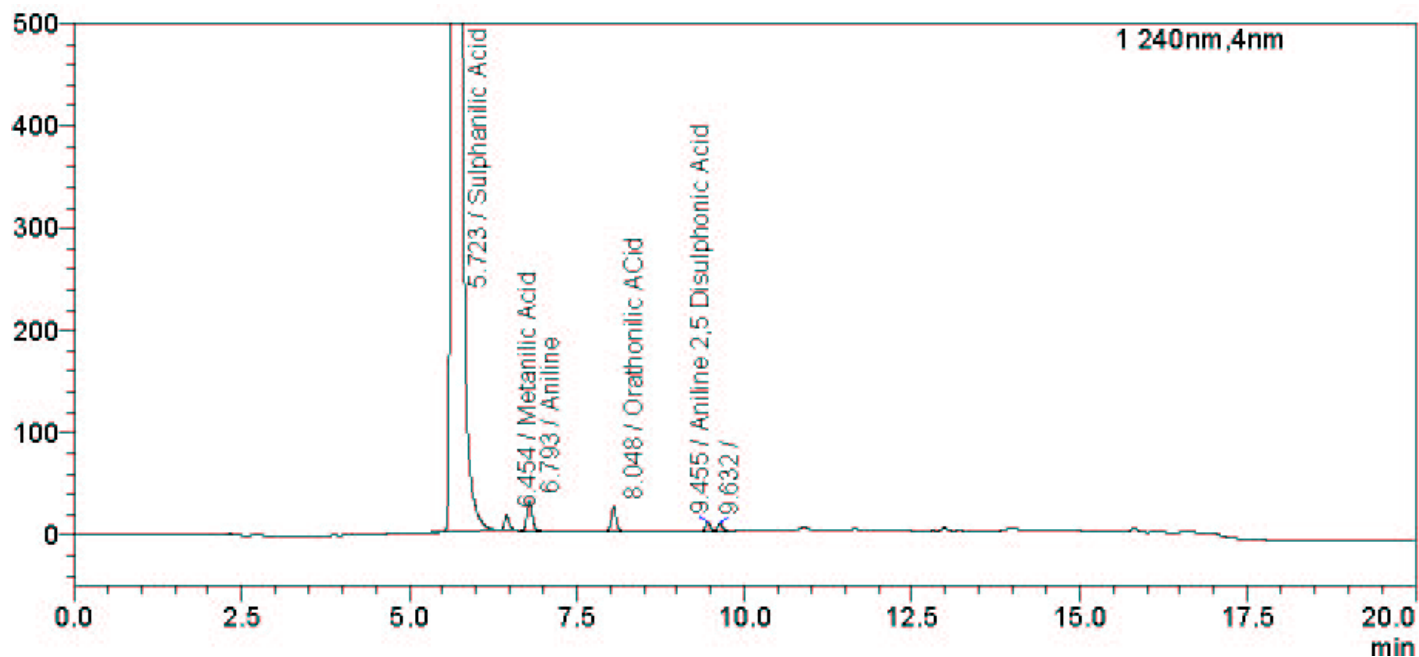
Aniline : 6.79 min

Orthanilic acid : 8.04 min

Aniline-2,5-disulphonic acid : 9.45 min

C-14 CALCULATION

Calculate the peak area of individual constituent pertaining to aniline/metanilic acid/orthanilic acid/aniline-2,5-disulphonic acid on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure aniline/metanilic acid/orthanilic acid/aniline-2,5-disulphonic acid.



$$\text{Aniline, percent} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where

A_1 = area of standard aniline;

V_1 = injection volume of standard aniline;

W_1 = weight of standard aniline;

B_1 = total volume of standard aniline;

A_1 = area of aniline peak in sample;

V_1 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.

Similarly, contents of metanilic acid, orthonilic acid, aniline-2,5-disulphonic acid shall be calculated.

ANNEX D*(Foreword)***COMMITTEE COMPOSITION**

Dye Intermediates Sectional Committee, PCD 26

<i>Organization</i>	<i>Representative(s)</i>
Institute of Chemical Technology, Mumbai	PROF GANAPATI SUBRAY SHANKARLING (Chairman)
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Colourtex Industries Ltd, Mumbai	DR PANKAJ DESAI SHRI R. K. JAISWAL (<i>Alternate</i>)
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Indian Beauty and Hygiene Association, Mumbai	MS MALATHI NARAYANAN
Indian Chemical Council, Mumbai	SHRI P. S. SINGH
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Member Secretary

Ms ADITI CHOUDHARY
SCIENTIST 'B' (PCD), BIS

Working Group for Validation of Test Methods, PCD 26/ WG-1

<i>Organization</i>	<i>Representative(s)</i>
Colourtex Industries Ltd, Mumbai	DR PANKAJ DESAI (<i>Convener</i>)
Aarti Industries Ltd, Mumbai	DR VAISHALI BHANDARY
Atul Limited, Gujarat	DR M. U. RAHMAN
Archroma India Pvt Ltd, Thane	DR RAJESH RAMAMURTHY
Central Revenue Control Laboratory, New Delhi	DR T. A SREENIVASA RAO
Heubach Colour Pvt Ltd, Mumbai	SHRI J. I. SEVAK
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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002

Telephones: 2323 0131, 2323 3375, 2323 9402

Website: www.bis.gov.in

Regional Offices:

	Telephones
Central : 601/A, Konnectus Tower-1, 6 th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	{ 2323 7617
Eastern : 8 th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	{ 2367 0012 2320 9474
Northern : Plot No. 4-A, Sector 27-B, Madhya Marg Chandigarh 160019	{ 265 9930
Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	{ 2254 1442 2254 1216
Western : Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	{ 2821 8093

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